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Dated 6 September 2000



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The Patent Office

Cardiff Road
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1. Your reference

P007518GB NJN TAG

2. Patent application number

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9920281.4

26 AUG 1999

3. Full name, address and occupation of each applicant
(underline all names)

COLLAG LIMITED
MAIDENSTONE HEATH
BLUNDELL LANE
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SO31 1AA

Patents ADP number (if you know it)

If the applicant is a corporate body, give the country/state of its incorporation

BRITISH

7005747001

4. Title of the invention

NOVEL COMPOSITIONS OF BIOLOGICALLY ACTIVE AGENTS AND THEIR USE

5. Name of your agent (if you have one)

"Address for service" in the United Kingdom to which all correspondence should be sent
(including the postcode)

Patents ADP number (if you have one)

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Country

Priority application number
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Date of filing
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Number of earlier application

Date of filing
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8. Is a statement of inventorship and of right to grant of a patent required in support of this request? (Answer 'Yes' if:
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Description **22**

Claims(s) **NONE**

Abstract **1**

Drawing(s) **NONE**

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Priority documents

Translations of priority documents

Statement of inventorship and right to grant of a patent (Patents Form 7/77)

Request for preliminary examination and search (Patents Form 9/77)

Request for substantive examination (Patents Form 10/77)

Any other documents (please specify)

11.

I/We request the grant of a patent on the basis of this application.

Signature

Date

D Young & Co.

D YOUNG & CO
Agents for the Applicants

26 August
1999

12. Name and daytime telephone number of the person to contact in the United Kingdom

NEIL NACHSHEN

0171 353 4343

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Novel Compositions of Biologically Active Agents and their Use

FIELD OF THE INVENTION

The invention relates to novel compositions of biologically active agents, which exhibit enhanced bio-availability on dilution in water and their use.

5 It is more particularly concerned with granular compositions of low dose-rate pesticides prepared by an extrusion process, which disperse rapidly in water to form stable suspensions of the active material, which subsequently dissolve at a rate and to an extent far higher than that achieved by conventional compositions. This property provides enhanced efficacy of the pesticides with attendant reduced crop
10 damage.

DESCRIPTION OF THE PRIOR ART

The advantages of dispersible granule formulations of pesticides are well known. Their ease of handling and reduced worker exposure compared to powder or liquid formulations are well documented. G. A. Bell, "Chemistry and Technology of 15 Agrochemical Formulations", Edited by D. A. Knowles (Kluver, 1998), pages 80-114, describes a range of dispersible granule types and processes for their manufacture.

The most popular process for preparing dispersible granules is extrusion. US 3,954,439 discloses granular compositions of a herbicidal agent and one or more 20 surfactants and processes for the production of such compositions. The process according to the invention is applicable to those herbicides, which are substantially insoluble in water. The patent states that it is obviously desirable that the granules should have the highest possible content of active herbicidal material. While the process according to the invention is preferably carried out so as to give granules 25 containing at least 50% of active herbicide material, it is more preferable that the granules should contain more than this, that is at least 80% and even up to 95%. The

patent also teaches that the total amount of surfactant should be minimized, preferably from 5-15%.

US 5,872,078 relates to dry, water soluble and/or water dispersible, agriculturally acceptable herbicidal compositions containing two or more herbicides, one of which is N-phosphonomethylglycine or acceptable water-soluble salt thereof. The water insoluble co-herbicide may be, amongst others, sulfonylureas such as Ally, Classic, Oust, Glean and mixtures thereof. A liquid surfactant is added to this mixture and extrusion granulation may be used to process the compositions described to form granules.

10 SUMMARY OF THE INVENTION

It has been surprisingly found that when low use-rate pesticides, normally water-insoluble, such as the sulfonyl ureas some of which are described in US 5,872,078, are intimately mixed in certain proportions with at least one high rate use pesticide and conventional other ingredients such as, a suitable surfactant or surfactants and processed into water-dispersible granules, the rate and extent of solubility and thus the bioavailability, of the active materials on dilution in water, at or below the normal use rate for the active, may be increased as compared to granules containing the equivalent amount of each material alone.

20 The advantages observed by the compositions of the present invention may therefore be described as synergistic, in that an effect greater than the combined additive affect of each individual component is observed.

25 The granular compositions are preferably prepared by the method described in UK Patent Application No. 9901479.7, the contents of which are hereby incorporated by reference.

The compositions of the present invention result in the individual components being used at lower rates and with less phytotoxicity than conventional compositions of the said active materials, against a wide range of pests and diseases.

5 DETAILED DESCRIPTION OF THE INVENTION

The invention comprises a dry, free-flowing, dustless and rapidly dispersing granular formulation containing a low use rate pesticide or mixture of pesticides together with an additional high use rate pesticide. The terms composition and formulation are used herein to have the same meaning.

10 A suitable surface active agent(s) may be incorporated into the formulation at a specific ratio so as to enable the rapid dispersion and subsequent dissolution of the low use rate and high use rate active material upon dilution and subsequent application.

15 The invention is particularly suitable for, but not limited to, such low use-rate pesticides as:

Abamectin, imidazolinone, azoxystrobin, bensulfuron-methyl, carfentrazone-ethyl, chlorsulfuron, cinosulfuron, clodinafop, clopyralid, lambda-cyhalothrin, 20 deltamethrin, diflufenican, emamectin benzoate, fibronil, flurtamone, imazamethabenz-methyl, imazapyr, imazethapyr, imadacloprid, metsulfuron-methyl, milbectin, nicosulfuron, pirimisulfuron-methyl, rimsulfuron, sulfometuron-methyl, thifensulfuron-methyl, tribenuron-methyl, and triflusulfuron-methyl.

25 Suitable high use rate pesticides include :

Ametryn, atrazine, benomylbentazone, bifenox, bromoxynil, captan, carbendazim, chloridazon, chlorothalonil, chlortoluron, lambda-cyhalothrin, cyhexatin,

cymoxynil, alpha-cypermethrin, deltamethrin, dimethomorph, diuron, ethofumesate, fibronil, flurtamone, glyphosate, imazamethabenz-methyl, imazapyr, imazethapyr, imadacloprid, isoproturon, linuron, mancozeb, maneb, metamitron, methiocarb, metribuzin, milbectin, oxadixyl, oxyfluorfen, phenmedipham, propanil, propyzamide, simazine, thifensulfuron-methyl and thiram.

In a preferred embodiment, the low rate use pesticide is bensulfuron-methyl and the high rate use pesticide is propanil.

10 Suitable surface active agents may include either wetting or dispersing agents or a combination of both.

Suitable wetting agents include: sodium alkyl aryl sulphonates, sodium alkyl aryl sulphosuccinates, sodium alkyl sulphates.

15 Suitable dispersing agents include : sodium lignosulphonates, sodium naphthalene sulphonate formaldehyde condensates, tristyrylphenol ethoxylate phosphate esters, aliphatic alcohol ethoxylates, alkylphenol ethoxylates, EO-PO block copolymers, "comb" graft copolymers, polyvinyl alcohol-vinyl acetate copolymers.

20 The mixture of components, either singularly or collectively, of the compositions described in this patent are first mixed in a suitable blender so that a uniform blend is obtained and then passed through a suitable milling system such as an air mill, pin mill or air-swept impact mill so that a fine powder (the pre-mix) comprising an average particle size of 0.5-20 microns, or more preferably between 0.5 – 5 microns is obtained. The powder thus obtained is agglomerated, so that uniform, dust-free granules are obtained, preferably by the process described in Patent Application No.

UK 9901479.7. This preferred method involves the extrusion of the wetted powder which is then in the form of a freely flowing homogeneous powder, in a low temperature, low pressure extruder as described in EP 812256B. The granules made by such a process are exemplified by their rapid dispersing properties on dilution in water and subsequent enhanced dissolution of low use rate actives contained in them.

The low use rate and high use rate pesticides may be combined in the formation of the dry pre-mix with the other formulation excipients or alternatively the pre-mix may be prepared with one of the pesticides and the other added to the milled pre-mix. This alternative approach is preferred when the high use rate pesticide is propanil, and the low use rate pesticide is then added to the pre-mix and blended with it prior to granulation.

This invention relates to the novel compositions and to methods of killing or controlling weeds by applying a reduced amount of the active ingredient(s), diluted in water, than that normally recommended for such active(s) against such weeds. In addition the invention allows for the avoidance of subsequent applications of the said actives, thus further reducing the amount of pesticide used.

The following examples are presented to illustrate the present invention as well as the various embodiments of the invention. These examples are presented as being illustrative of the novel formulations, process for preparing the invention and pesticidal use thereof and are not intended to be a limitation of the scope of this invention.

EXAMPLES**Example 1****Chlorsulfuron 75 WG**

	Ingredient	Trade name	% w/w
5	Chlorsulfuron technical (95%) (technical a.i.)		78.95
	Sodium lignosulfonate	Ultrazine NA	12.50
	Di isopropyl naphthalene sulfonate, sodium salt	Galoryl MT704	1.00
10	Lactose	Lactose	7.55

Method

The chlorsulfuron technical was airmilled using a Gem-T airmill before
 15 combining with other components.

The milled chlorsulfuron technical, Ultrazine and Galoryl components were
 blended until uniform in a high speed blender. The lactose was then added and the
 formulation blended for a further 15 seconds. 17% distilled water was added
 20 whilst blending. The wetted premix (free flowing powder) was fed to a basket
 extruder as described in EP812256B through a 1mm screen. A compacted
 extrudate was obtained and the resulting granules dried at 60C for 8 minutes. The
 dried granules were then sieved through 2 mm and 500 micron sieves.

Example 2**Chlorsulfuron 25 WG**

Ingredient	Trade name	% w/w
5 Chlorsulfuron technical (95%)	(technical a.i.)	26.32
Sodium lignosulfonate	Ultrazine NA	12.50
Dodedyl benzene sulphonate,	Arylan SX85	5.00
Sodium salt		
Lactose	Lactose	56.18

10

Method

The chlorsulfuron technical was airmilled using a Gem-T airmill before combining with other components.

15

The technical, Ultrazine and Arylan components were blended until uniform in a high speed blender. The lactose was then added and the formulation blended for a further 15 seconds. 12% distilled water was added whilst blending. The wetted premix (free flowing powder) was fed to a basket extruder as described in EP812256B through a 1mm screen. A compacted extrudate was obtained and the resulting granules dried at 60C for 8 minutes. The dried granules were then sieved through 2 mm and 500 micron sieves.

20
25 The solubility of the active in the above formulations, Glean (commercial 75 WG product) and airmilled technical was tested using the method below:

Solubility test method

200 mls water was poured into a jacketed glass vessel and allowed to reach 25C. A Grant recirculator was used to maintain the temperature at 25C +/- 1C. A magnetic stirrer at a set speed was used to stir the water. The specified weight of granules was then added to the water and allowed to disperse for 30 seconds before a timer was started. A 2 mls sample was removed using a syringe after 5 minutes and filtered using a 0.45 micron syringe filter. The solution was then analysed to determine the active concentration using a HPLC method.

5

10 The theoretical concentration assuming 100 % solubility was calculated using an assay obtained using the HPLC.

The following data was obtained:

Formulation	Product	Dilution rate (mgs a.i. / L)	Type of water used to dilute granules	% active added to water that dissolved after 5 minutes
Example 1	75 WG	72	Distilled	95
Example 2	25 WG	69	Distilled	98
Glean (Du Pont)	75 WG	72	Distilled	67
Airmilled technical	-	91	Distilled	< 2

Conclusions

Both the 75 and 25 extruded WG formulations have a significantly higher solubility in distilled water compared to the commercial product.

5

The technical is not readily soluble in distilled water at this temperature.

Example 3

Bensulfuron 60 WG

10

Ingredient	Trade name	% w/w
Bensulfuron-methyl technical (95%)	(technical a.i.)	64.21
Naphthalene sulfonic acid	Galoryl DT505	12.70
Formaldehyde condensate, sodium salt		
15 Di isopropyl naphthalene sulfonate, sodium salt	Galoryl MT704	1.00
Lactose	Lactose	22.08

Example 4

Bensulfuron 1 WG

Ingredient	Trade name	% w/w
Bensulfuron methyl technical (95%)	(technical a.i.)	1.05
Naphthalene sulfonic acid	Galoryl DT505	12.70
Formaldehyde condensate, sodium salt		
25 Di isopropyl naphthalene sulfonate, sodium salt	Galoryl MT704	1.00
Lactose	Lactose	85.25

Method

The technical and Galoryl DT505 were blended together until uniform. The blend

5 was then airmilled using a Gem-T airmill.

The milled premix, Galoryl MT704 and lactose were blended until uniform in a high speed blender. The lactose was then added and the formulation blended for a further 15 seconds. 12% distilled water was added whilst blending. The wetted 10 premix (free flowing powder) was fed to a basket extruder as described in EP812256B through a 1mm screen. A compacted extrudate was obtained and the resulting granules dried at 60C for 8 minutes. The dried granules were then sieved through 2 mm and 500 micron sieves. The solubility was then tested using the method detailed in example 1.

15 The following data was obtained:

Formulation	Product	Dilution rate (mgs a.i. / L)	Type of water used to dilute granules	% active added to water that dissolved after 5 minutes
Londax	60 WG	300	Tap	12
Example 3	60 WG	291	Tap	25
Londax	60 WG	75	Tap	19
Example 3	60 WG	73	Tap	46
Example 4	1 WG	240	Tap	71

Conclusions

There is a significant difference between Londax and Example 3 at both rates tested. The solubility is highly dependent on both the dilution rate and the active ingredient level.

Further solubility testing up to 2 hours was carried out using the same method as for example 1 but 1000 mls water was used to dilute the granules. Samples were taken after 5-minutes, 30 minutes, 1 hour, 1.5 hours and 2 hours:

The following results were obtained using 150 mgs a.i. / litre (all in tap water):

Time (mins)	% active added to water that dissolved	
	Example 3	Londax
5	26	12
30	30	21
60	34	26
90	37	30
120	39	34

Example 5**Propanil and bensulfuron combined WG (75 % propanil and 0.75 % bensulfuron)****5 Propanil Premix**

A premix of Propanil was prepared as follows:

Ingredient	Trade name	% w/w
Propanil technical (97.0% a.i.)	technical	82.47
10 Starch	Paselli	1.00
Nonionic surfactant and sodium lignosulphonate blend	Stepsperse DF 500	5.00
Modified sodium lignosulphonate	Ufoxane 3A	5.00
Hydrated aluminium silicate	China Clay	to 100

15

Method

The ingredients were blended in a medium shear, high speed blender for 5 minutes until uniform. The resulting mixture was passed through an air mill to obtain a fine powder. The powder was wetted with 19.5 % water (based on the dry weight of powder) and blended until a damp free flowing powder was formed. The premix was used in the following blends with bensulfuron:

Ingredient	Trade name	% w/w
Bensulfuron methyl technical (95%)	(technical a.i.)	0.79
Propanil 80% milled premix		93.75
25 Naphthalene sulfonic acid	Galoryl DT505	0.31
formaldehyde condensate, sodium salt		
China clay	China clay GTY	5.15

Method

The technical and Galoryl DT505 were blended together until uniform. The blend was then airmilled using a Gem-T airmill.

5

The milled bensulfuron and milled propanil premixes, Galoryl MT704 and china clay were blended until uniform in a high speed blender. The lactose was then added and the formulation blended for a further 15 seconds. 17 % distilled water was added whilst blending. The wetted premix (free flowing powder) was fed to a basket extruder as described in EP812256B through a 1mm screen. A compacted extrudate was obtained and the resulting granules dried at 60C for 8 minutes. The dried granules were then sieved through 2 mm and 500 micron sieves.

Example 6

15

Propanil and bensulfuron combined WG (75 % propanil and 0.375 % bensulfuron)

Ingredient	Trade name	% w/w
Bensulfuron methyl technical (95%)	(technical a.i.)	0.39
Propanil 80% milled premix		93.75
Naphthalene sulfonic acid	Galoryl DT505	0.16
formaldehyde condensate, sodium salt		
China clay	China clay GTY	5.70

Method

5 The technical and Galoryl DT505 were blended together until uniform. The blend was then airmilled using a Gem-T airmill.

10 The milled bensulfuron and milled propanil premixes, Galoryl MT704 and china clay were blended until uniform in a high speed blender. The lactose was then added and the formulation blended for a further 15 seconds. 17% distilled water was added whilst blending. The wetted premix (free flowing powder) was fed to a basket extruder as described in EP812256B through a 1mm screen. A compacted extrudate was obtained and the resulting granules dried at 60C for 8 minutes. The dried granules were then sieved through 2 mm and 500 micron sieves.

15

The above combination formulations were tested using the solubility method detailed in example 1. The following data was obtained:

Formulation	% bensulfuron a.i.	Dilution rate (mgs a.i. / L)	Type of water used to dilute granules	% active added to water that dissolved after 5 minutes
Example 5	0.75	75	Tap	66
	0.75	112.5	Tap	64
Example 6	0.375	37.5	Tap	83
	0.375	56	Tap	84

Further solubility testing up to 2 hours was carried out using the same method as for example 3.

The following results were obtained using 37.5 mgs a.i. / litre (all in tap water).

Data for Example 3 (bensulfuron 60 WG) and Londax (commercial bensulfuron 5 60 WG) at the same dilution rate is shown for comparison.

Time (mins)	% active added to water that dissolved		
	Example 5	Example 3	Londax
5	67	42	15
30	67	52	32
60	67	57	39
90	66	57	49
120	69	62	53

Conclusions

10 The solubility rate of bensulfuron in a combination granule containing an active that is used a high rate per hectare, is significantly higher compared with diluting the bensulfuron as a 60 WG.

Example 7**Propanil and bensulfuron combined WG (75 % propanil and 0.24 % bensulfuron)**

	Ingredient	Trade name	% w/w
5	Bensulfuron methyl technical (95%)	(technical a.i.)	0.25
	Propanil 80% milled premix		93.72
	Naphthalene sulfonic acid formaldehyde condensate, sodium salt	Galoryl DT505	0.10
10	China clay	China clay GTY	5.90

Method

The standard processing method was employed, with the bensulfuron being milled as a premix with the Galoryl DT505.

15 The solubility of the bensulfuron in the above formulation was then tested using the method detailed above.

The following results were obtained using 25 mgs and 31.3 mgs bensulfuron a.i. / litre (in tap water).

Time (mins)	% bensulfuron active added to water that dissolved	
	Example 7 (25 mgs /L)	Example 7 (31.3 mgs / L)
5	89	91
30	90	92
60	94	92
90	93	96
120	97	95

Field Evaluation

A composition according to Example 6 was evaluated in the field in comparison with commercial formulations containing the same active ingredients.

5 Treatment Details

Number	Treatment Composition	Rate of use (g / Hectare) Product
Control	Untreated Control	-
1	Stam 80 EDF	4,000g at Growth Stage BBCH 12-13 8,000g at Growth Stage BBCH 21-25
2	Stam 80 EDF + Londax 60	4,000g at Growth Stage BBCH 12-13 100g at Growth Stage BBCH 12-13
3	Example 6	4,000g at Growth Stage BBCH 12-13

The above treatments were applied in 400 l water/ha on Rice *v.loto* against Gramineae family weeds. The weeds were assessed at -1, +4, +16 and +32 days after application by the efficacy assessment guidelines provided by EPPO Guidelines PP1/181(2), PP1/152(2) and 1/62(2).

Stam 80 EDF is a commercial formulation containing 80% propanil in the form of an extruded granule.

Londax 60 is a commercial formulation containing 60% bensulfuron in the form of a fluid bed granule.

Results

Assessment : 1 day before 1st Application

Weeds	Control	Treatment 1			Treatment 2			Treatment 3		
		% cov.	% cov	% eff.	Sym pt.	% co v.	% eff.	Sy mpt	% cov.	% eff.
<i>Heteranthera limosa</i>	38.0	37.0	0.0	n.a.	35.0	0.0	n.a.	35.0	0.0	n.a.
<i>Heteranthera reniformis</i>	0.0	1.0	0.0	n.a.	0.0	0.0	n.a.	0.0	0.0	n.a.
<i>Echinochloa crus-galli</i>	1.0	1.0	0.0	n.a.	1.0	0.0	n.a.	1.0	0.0	n.a.
<i>Panicum dichotomiflorum</i>	1.0	1.0	0.0	n.a.	1.0	0.0	n.a.	1.0	0.0	n.a.
<i>Scirpus maritimus</i>	1.0	1.0	0.0	n.a.	1.0	0.0	n.a.	2.0	0.0	n.a.
<i>Scirpus mucronatus</i>	1.0	1.0	0.0	n.a.	2.0	0.0	n.a.	1.0	0.0	n.a.

Assessment : 4 days after 1st Application

Weeds	Control 1	Treatment 1			Treatment 2			Treatment 3		
		% cov.	% cov.	% eff.	Sym pt.	% cov.	% eff.	Sy mpt	% cov.	% eff.
<i>Heteranthera limosa</i>	55.0	55.0	70.0	WC	34.0	80.0	W	40.0	50.0	W
<i>Heteranthera reniformis</i>	0.5	0.0	0.0	n.a.	0.6	0.0	n.a.	0.0	0.0	n.a.
<i>Echinochloa crus-galli</i>	7.0	0.0	0.0	n.a.	1.0	0.0	n.a.	1.0	0.0	n.a.
<i>Panicum dichotomiflorum</i>	5.5	0.0	0.0	n.a.	1.0	0.0	n.a.	1.0	0.0	n.a.
<i>Scirpus maritimus</i>	1.0	4.0	90.0	W	0.0	0.0	n.a.	3.0	70.0	W
<i>Scirpus mucronatus</i>	2.0	1.0	60.0	W	2.0	60.0	W	5.0	70.0	W

Assessment : 16 days after 1st Application

Weeds	Control 1	Treatment 1			Treatment 2			Treatment 3		
		% cov. v.	% eff.	Sym pt.	% cov	% eff.	Sym pt.	% cov v.	% eff.	Sym pt.
<i>Heteranthera limosa</i>	55.0	1.0	99.0	W.C.	18.0	80.0	W.C.	2.0	98.0	W
<i>Heteranthera reniformis</i>	0.0	0.0	0.0	n.a.	0.0	0.0	n.a.	0.0	0.0	n.a.
<i>Echinochloa crus-galli</i>	10.0	0.0	0.0	n.a.	1.0	0.0	n.a.	0.0	0.0	n.a.
<i>Panicum dichotomiflorum</i>	15.0	0.0	0.0	n.a.	1.0	0.0	n.a.	0.0	0.0	n.a.
<i>Scirpus maritimus</i>	5.0	0.0	0.0	n.a.	0.0	0.0	n.a.	1.0	98.0	W
<i>Scirpus mucronatus</i>	15.0	0.0	0.0	n.a.	0.0	0.0	n.a.	0.0	0.0	n.a.

Assessment : 32 days after 1st Application

Weeds	Control	Treatment 1			Treatment 2			Treatment 3		
		% cov.	% cov.	% eff.	Sympt.	% cov.	% eff.	Sympt.	% cov.	% eff.
<i>Heteranthera limosa</i>	37.0	0.0	0.0	n.a.	1.0	0.0	n.a.	0.0	0.0	n.a.
<i>Heteranthera reniformis</i>	1.0	0.0	0.0	n.a.	0.0	0.0	n.a.	0.0	0.0	n.a.
<i>Echinochloa crus-galli</i>	10.0	0.0	0.0	n.a.	0.0	0.0	n.a.	0.0	0.0	n.a.
<i>Panicum dichotomiflorum</i>	15.0	0.0	0.0	n.a.	2.0	0.0	n.a.	0.0	0.0	n.a.
<i>Scirpus maritimus</i>	5.0	0.0	0.0	n.a.	0.0	0.0	n.a.	0.0	0.0	n.a.
<i>Scirpus mucronatus</i>	32.0	0.0	0.0	n.a.	0.0	0.0	n.a.	0.0	0.0	n.a.

5 Abbreviations :**% cov. : % area covered by weeds****% eff. : % herbicide efficacy (% of weeds showing necrotic symptoms):****Sympt. : symptoms (W: withered; C:chlorotic; n.a. not applicable)**

Conclusions

The above results demonstrate that Example 6 (15g/ha bensulfuron combined with 3,000 g/ha propanil) provides equivalent control to the commercial products tested when applied at less than $\frac{1}{3}$ rd of the propanil in the Stam 80 EDF treatment (two applications, one 3200 a.i.g/ha and one 6,400 a.i. g/ha propanil) and $\frac{1}{4}$ of the Londax rate (one application of 60g/ha bensulfuron tank mixed with one application of 3,200 g/ha propanil)

ABSTRACT

The invention relates to novel compositions of biologically active agents, which exhibit enhanced bio-availability on dilution and application in water.

